



Long period grating based toluene sensor for use with water contamination



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ABSTRACT

A demonstration of the use of a calix[4]resorcinarene coated optical fibre long period grating sensor for the detection of toluene in water is presented. Monitoring water quality both for domestic use and around industrial sites is critical to the preservation of clean water provision. Here we show that, by using a fibre optic based sensor system, water quality monitoring can be carried out without the requirement for water sampling or pre-concentration. The results presented demonstrate that this proof-of-concept sensor is capable of sensing ~100 ppm concentrations of toluene with semi-selectivity and low (<10 ppm) variation.

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1. Introduction

Recently, optical fibre long period gratings (LPGs) coated with nanoscale films of chemical recognition molecules have been investigated as a platform for chemical sensing. Sensors for a range of analytes in liquid and gaseous environments have been demonstrated, detecting, for example, aromatic carboxylic acids [1,2], cadmium [3,4] and ethanol vapour [5,6]. Appropriate choice of grating period and optical thickness of the coating can result in high sensitivity, for example a limit of detection of 140 ppb for ammonia has been reported [1,7].

The development of sensors for volatile organic compounds (VOC) is an area of significant interest, facilitating the monitoring of air quality in indoor and outdoor environments, and the monitoring of soil and water contamination. The toxicity of the BTEX chemicals, benzene, toluene, ethylbenzene and xylene makes their sensing of particular significance. Calixarenes have been investigated as functional coatings for VOC sensing using LPGs [3], where they are used as molecular 'buckets' to form temporary solid, liquid and gas state complexes. Depending on the calixarene used, only certain molecules with a particular charge and size will form complexes

with the coating. These complexes can be formed from a great many types of interaction, such as hydrogen bonding or electrostatic. In addition to complex formation, calixarenes form a natural cavity with an approximate diameter of around 1–2 nm. These small cavities encourage the condensation of the target vapour below the natural condensation point of the vapour. In the case of calixarenes it has been shown previously that this can be as low as 0.2 Ps (Ps is the saturated vapour pressure) [5]. This condensation within the film causes a swelling and an optical thickness change, the degree of which correlates highly with the mobility of the vapour through the calixarene layers [3,7].

As identified in [3], it is the swelling of the calixarene film that is being sensed via the LPG, rather than the uptake of the analyte into a gas state complex. This compromises the sensitivity and limit of detection of VOC vapour sensors based on calixarene coated LPGs, as only a limited number of calixarene molecules would interact with the vapour. For example, at 10 ppm less than 1–2% of sites would be interacting with the target BTEX gas as determined from simple modeling of the sensor interactions. Previous work by Hassan et al. [5,8] focused on measuring film reaction using ellipsometry and surface plasmon resonance (SPR). This work demonstrated that, at this concentration, the refractive index change was smaller than the uncertainty (refractive index of ± 0.001) of the measurement systems. This limits the sensor's suitability for BTEX gas sensing, which would require a sensitivity of around 10–100 ppm to be of practical use. However, the number of molecules present

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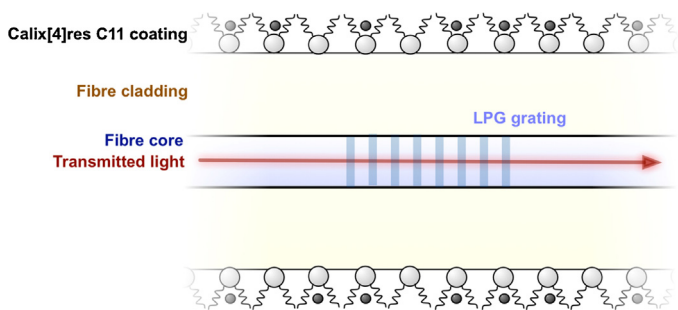


Fig. 1. Schematic of the optical fibre sensor.

at a given concentration is larger in solution than it is in a vapour. Thus the calixarene coated LPG may have appropriate sensitivity when used to detect BTEX chemicals in solution. Here we expand on the work presented in [3,9] and demonstrate that this gas sensor system can be adapted to work in an aqueous environment for use in environmental monitoring.

The calix[4]res C11 molecule primarily used in this work and prior work has limited selectivity and has been shown to react to a wide range of aromatic hydrocarbons [3,10]. In previous reports it has been suggested that this poor selectivity could be corrected with pre-concentration stages or pre-filtration step upstream from the sensor [8,11]. However, when used as a sensor in an aqueous environment, this poor selectivity can be an advantage in the detection of contamination such as oil spills. Crude oil comprises 3 key groups of chemicals; paraffins, naphthalenes and other aromatic hydrocarbons. The ratio of these varies depending on the age of the crude oil [9,12] but, on average, crude oil contains around 30% aromatic hydrocarbons. Recent environmental disasters in the Gulf [3,10] have highlighted the need to find new ways of tracking and monitoring the movement of oil spills. A large percentage of the oil that leaked into the environment was trapped in undersea currents and could not be tracked prior to washing up in nature reserves and on beaches. Having a sensor system for finding these spills would be highly beneficial and would help reduce the environmental impact of such disasters. A fibre optic sensor coated with calix[4]res C11 is a good candidate for this application as firstly the fibre optic sensor and the calixarene can survive the highly variable conditions of use in the sea [3,11], and secondly the partial selectivity of calix[4]res C11 makes it ideal for detecting the presence of mixes of aromatic hydrocarbons, enabling the sensor to detect the presence of crude oil at far lower concentrations due to the cumulative effect of the mixed aromatics in the oil. Finally, while calix[4]res C11 has no selectivity between aromatic hydrocarbons [12–14], it has been shown to have little to no response to other possible contaminants such as alcohols [3,15], which may be more closely associated with organic decay.

A diagram of the sensor is shown in Fig. 1.

An optical fibre LPG comprises a periodic modulation of the refractive index of the core of an optical fibre, typically induced by exposure to the output from a UV laser. LPGs act to couple the light between co-propagating core and cladding modes of an optical fibre at discrete wavelengths, which results in a transmission spectrum that consists of a series of resonance bands. The central wavelengths of the resonance bands are determined by the product of the period of the LPG with the difference between the core and cladding mode indices. The sensitivity of the effective index of the coupled modes to their external surroundings manifests itself as a change in the central wavelength and/or depth of the resonance bands and allows the use of LPGs to sense parameters such as temperature, strain, pressure, bending and surrounding refractive index. LPGs have also been used for multi-parameter sensing as the numerous resonance bands in the transmission spectrum

show different sensitivities to the various environmental changes and can therefore be used to monitor multiple parameters at the same time.

2. Methods and materials

An LPG of period 97 μm and of length 35 mm was fabricated in a photosensitive single-mode fibre with a cut-off wavelength of 627 nm (Fibercore PS750) using the point-by-point method. The fibre was attached onto a high-resolution translation stage and positioned behind a slit with a width equal to half the desired period. The fibre was irradiated using a UV laser beam at 266 nm from an injection-seeded, frequency quadrupled Spectra Physics Quanta-Ray Nd:YAG laser through the slit. The computer controlled translation stage dwelled at a given position for 30 s while a section of fibre was exposed to the UV radiation and then moved the optical fibre by the desired period, dwelling for 30 s. The process was repeated until the LPG had reached the desired length.

The fibre was coated using the Langmuir Blodgett (LB) technique, in which the film is deposited molecular layer by molecular layer. As in [3,16], the material used here was calix[4]res C11, which has been shown previously to be sensitive to a range of aromatic hydrocarbons. Prior to the deposition of the coating, the fibre was cleaned with chloroform and an IPA soaked optical tissue and mounted on the dipper of a Nima Alternate layer trough. This trough was subsequently filled with de-ionized water and 50 μl of a 1 mg ml^{-1} chloroform solution of calix[4]res C11 was added to the surface. The material was allowed to spread for 30 min before being compressed to a surface pressure of 37.5 mN m^{-1} , and then held at this pressure for 1 h to ensure that the film had stabilized fully. This surface pressure was chosen as previous work [3,14] had shown that this pressure facilitates a bilayer deposition of calix[4]res C11 on glass surfaces, which was also confirmed by our own studies (not presented here). During the coating deposition, the fibre was moved up and down through the monolayer at a speed of 15 mm s^{-1} and the transmission spectrum of the fibre was recorded at the bottom of each dip cycle, with the coated section of the fibre fully immersed in water, using an Ocean Optics S2000 spectrophotometer. The fibre was coated with 190 bilayers (380 cycles up and down through the monolayer) of calix[4]res C11. A grey scale plot showing the evolution of the spectral properties of the LPG as a function of layer deposition is shown in Fig. 2.

The white bands in the grey scale plot (Fig. 2) show the movement of the resonance bands in the LPG transmission spectrum

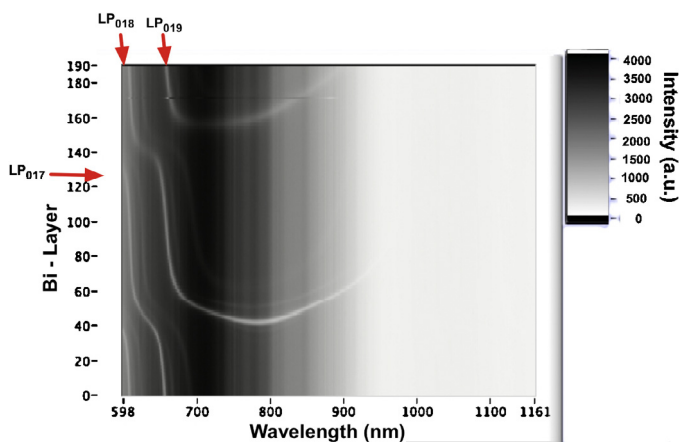


Fig. 2. Grey scale plot of the transmission spectrum (recorded in solution) of an LPG of period 97 μm , fabricated in an optical fibre of cut off wavelength 670 nm, during LB deposition of a coating of calix[4]res C11.

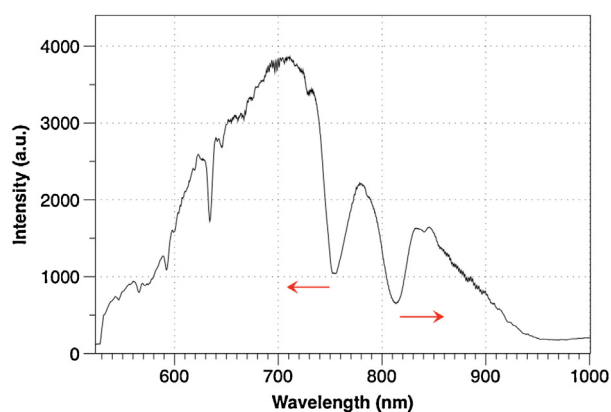


Fig. 3. Transmission spectrum of an LPG of period $97\ \mu\text{m}$, fabricated in an optical fibre of cut off wavelength $670\ \text{nm}$, after LB deposition of 56 layers of calix[4]res C11. The arrows indicate the direction of the wavelength shift of the dual resonance bands in response to increasing optical thickness of the coating.

in response to increasing coating thickness. The plot illustrates a number of significant features of coated LPGs. Focusing on the resonance bands labelled, LP_{017} , and LP_{018} , for numbers of coated bi-layers between 40 and 45 the resonance wavelengths show high sensitivity to changes in the optical thickness of the coating. This region, termed the mode transition region, corresponds to the coating thickness at which one of the cladding modes is coupled into the coating, requiring a reorganization of the cladding modes [3,15], which causes significant changes in the cladding modes' refractive indices with concomitant changes in the resonance conditions. The second feature concerns the resonance band labelled LP_{019} , which shows the development of a resonance band at a wavelength of approximately $790\ \text{nm}$ for 38 bi-layers. With increasing optical thickness of the coating, the band splits into two. This effect is related to the properties of the phase matching condition, which contains a turning point. Selection of the period of the LPG such that LPG operates at the phase matching turning point for particular cladding modes ensures maximum sensitivity to environmental perturbation. In the case of coated LPGs, optimum sensitivity is achieved when the phase matching turning point and the mode transition region coincide [3,16], which requires appropriate selection of the grating period and of the coating's optical thickness, taking into consideration the refractive index of the medium in which the LPG will be immersed and the likely operating temperature. Achieving this is further complicated by the inevitable uncertainties in the refractive indices of the core and cladding of the fibre. Based on the characterisation of the evolution of the transmission spectrum of the LPG with increasing coating thickness shown in Fig. 2, we decided to operate near the phase matching turning point and within the mode transition region, such that the analysis of the spectrum involved the measurement of changes in the separation of the dual resonance bands corresponding to coupling to the LP_{019} mode. Thus there is scope to improve the sensitivity of the device by fabricating an LPG with a different period, which would require sub-micron control of the period [17,18]. Following the characterisation of the coating process, the fibre was cleaned using chloroform and IPA soaked optical wipes to remove the coating. The effectiveness of the cleaning process was confirmed by comparison of the spectra before coating to the spectra of the coated and cleaned fibre. Once cleaned and re-mounted in the LB trough, the fibre was coated with 56 layers using the previously described coating method to bring the operation of the device into the most sensitive region. The resulting spectrum of the fibre is shown in Fig. 3. The two arrows shown in Fig. 3 indicate the direction in which the two resonance bands would shift in response to a change

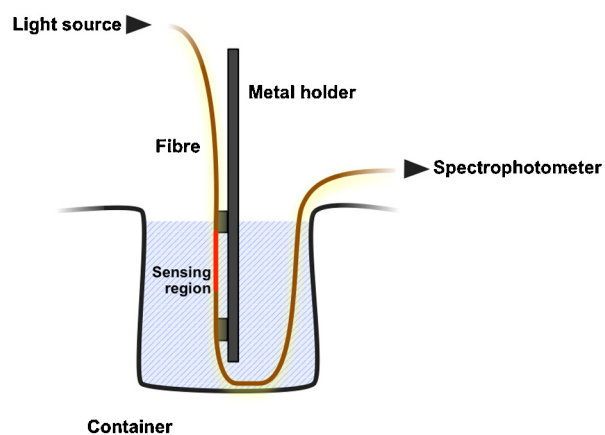


Fig. 4. Experimental setup.

in the optical thickness of the coating. In the results presented in this paper, the separation of the resonance bands is plotted.

Once coated, the optical fibre was mounted in a square container, in which it could be immersed in a range of solutions, as shown in Fig. 4.

To investigate the response time of the sensor, the LPG was immersed in a volume of $480\ \text{ml}$ of water, and a volume of $10\ \text{ml}$ of toluene was added to the water. The response of the transmission spectrum occurred within the integration time of the CCD spectrometer, and thus the response time was better than $400\ \text{ms}$ even when the toluene concentration was not uniform in the solution. To ensure consistency in the characterisation of the sensor's sensitivity to toluene, all subsequent experiments were performed by filling the surrounding container (volume $480\ \text{ml}$) with a pre-mixed solution of known concentration, and recording the spectrum of the fibre after a 5-min stabilisation period. 5-min was chosen as this allowed movement in the surrounding water to lessen any interference effect from fluid movement on the fibre. Following each experiment, the tank was emptied and rinsed with de-ionized water before to adding the next solution. All experiments were carried out at $22.5\ ^\circ\text{C}$ in a dust free temperature controlled room. The solution temperature was confirmed prior to use.

3. Results

The stability of the sensor within the experimental setup was investigated. One concern was that the repeated washing of the sensor system may degrade the coating and reduce the sensitivity over time. An examination of the sensor's stability was conducted over 3 days with a number of repeat measurements being made on each day. In each experiment the container was filled with de-ionized water, the spectra recorded and the container was subsequently washed with 2–3 refills of water before being re-filled with the appropriate test solution and the next spectra recorded.

In Fig. 5 there appears to be a shift from day 1 to day 2 and 3, however when examined using an ANOVA statistical test this difference was found to be statistically insignificant ($p < 0.05$).

The sensor was then exposed to a range of toluene dilutions (0, 1, 10, 50, 100, 200 and $400\ \text{ppm}$ by mass). These dilutions were prepared by adding an appropriate volume of pure toluene to deionized water; the combined solution was then shaken vigorously and sonicated for $30\ \text{s}$ to ensure that the toluene had dissolved in the water. Of the BTEX compounds only toluene was used, as from the work by Topliss et al. [3] and Hassan et al. [20] it can be seen that calix[4]res reacts equally to these materials and would be suitable for proof-of-principle testing.

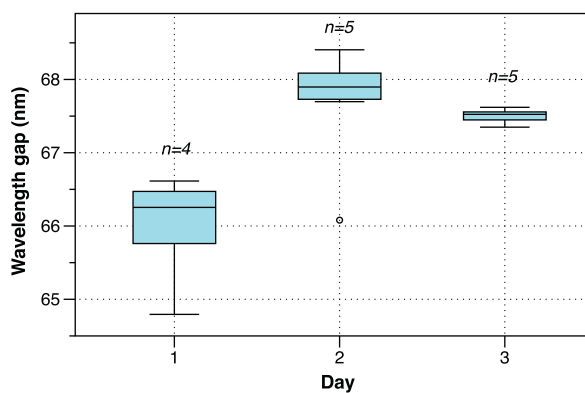


Fig. 5. Box and whisker plot showing the stability of the sensor (measured by wavelength separation) during repeats measurement of de-ionized water samples over 3 days.

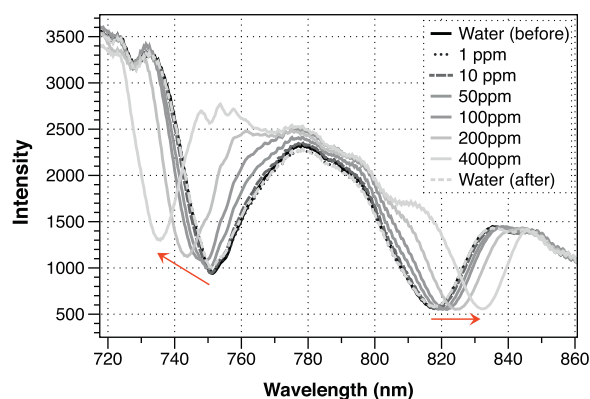


Fig. 6. Transmission spectra of the coated LPG sensor in response to exposure to a range of toluene dilutions. The arrows indicate the movement of the dual resonance bands in response to increasing toluene concentration.

As with previous experiments, the toluene solutions were then poured into the vessel surrounding the fibre and the spectra was recorded after a 5 min settling period. The resulting spectra, recorded over a spectral range of 720–845 nm, are shown in Fig. 6. The separation of the dual resonance bands increases with increasing concentration of toluene. Included in Fig. 6 are two measurements for pure water taken before and after the dilution curve was run to demonstrate the reversibility of the sensor system.

It has been reported previously [12] for calix[4]res C11 that the sensor is specific to aromatic hydrocarbons. The same sensor was subsequently exposed to a range of ethanol dilutions (0, 1, 10, 50, 100, 200 and 400 ppm by mass) in order to demonstrate that, as has been reported previously for calix[4]res C11, the sensor was specific to aromatic hydrocarbons and was not affected by other possible contaminants that may be present in water samples [3]. Ethanol was chosen, as alcohols are one possible natural contaminant within any aromatic hydrocarbon sensing. This data is presented alongside the toluene dilutions data in Fig. 7, showing a linear relationship between the wavelength separation of the dual resonance bands and the concentration of toluene across this range explored. Ethanol by comparison shows no measurable response, indicating that the system is semi-specific to toluene over ethanol as suggested in [3].

The limit of detection (LOD) and limit of quantification (LOQ) can be determined from the linear fit. The LOD is estimated to be 3 times the standard deviation (LOD = 41 ppm) and the LOQ is estimated to be 10 times the standard deviation (LOQ = 137 ppm).

In order to demonstrate the accuracy of these limits, a further experiment in which several repeat ($n=6$) measurements

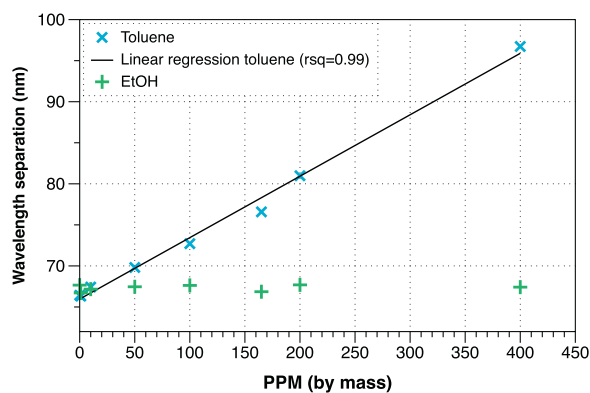


Fig. 7. Dilution curve of Toluene and Ethanol solution and their response of the calix[4]res C11 coated LPG sensor ($n=1$).

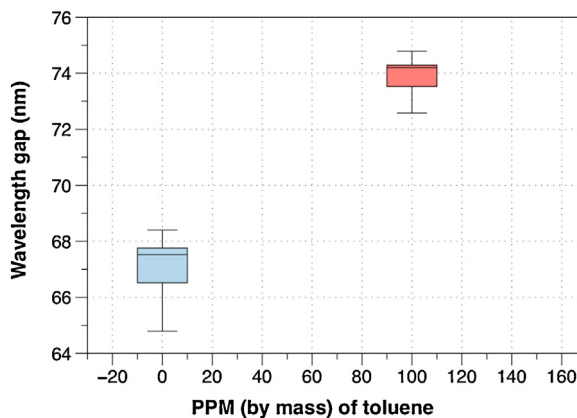


Fig. 8. Box and whisker plot of the sensor readings while immersed in water compared with immersion in 100 ppm toluene solution ($n=6$).

were taken for a 100 ppm toluene solution. This concentration was chosen as it fell between the LOD and LOQ limits. As with the experiments undertaken in distilled water described previously, the trough was emptied and re-filled with a fresh solution between each measurement. The measured wavelength separation is compared to the data from Fig. 8.

As predicted by the LOD and LOQ calculations the two runs are significantly different as determined by a non-paired t -test ($p < 0.05$). This demonstrates that the system can differentiate toluene in water concentrations of 100 ppm from background levels.

4. Conclusion

An LPG coated with a thin film of calix[4]res C11 via the LB technique, capable of detecting toluene in an aqueous solution with semi-selective sensitivity to dissolved toluene down to ppm levels, has been described. This performance is superior to the kppm levels detected in previous vapor phase work [3]. This sensor could be used for the detection of contamination in water. The sensor discussed has a limit of detection of ~ 100 ppm; which is close the target range set for oil weep sampling [9,19] and levels seen around leaking oil plumes [20–22]. Further work will need to focus on other possible cross-reactive species and improving the variance of the system.

It is also expected that, with further optimisation particularly of the period and coating thickness, this sensor platform could also be adapted for use for applications with higher sensitivity requirements, such as drinking water testing which requires a limit of detection of around 0.5–1 ppm [23].

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.snb.2014.06.121>.

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Biographies

Dr Matthew Partridge currently works as a research fellow within Engineering Photonics at Cranfield University. He also completed his Ph.D. at Cranfield University in a joint project between Engineering Photonics and Cranfield Health. He has worked extensively in sensor development, including immunoassays, micro arrays, microfluidics and optical sensors. His current research area is primarily focused on combining novel photonic techniques with Langmuir monolayers to develop improved sensor systems. In addition he also runs an open science blog (errantscience.com) aimed at disseminating complex science to a wider audience.

Rebecca Y N Wong is currently completing her Ph.D. on optical fibre long period grating sensors at Cranfield University. She received her B.Sc. in Physics from Imperial College London in 2009 and her M.Sc. in Photon Science from the University of Manchester in 2010.

Dr Stephen James was born and bred in Llanelli, Wales. He gained an MSc in 1988 in Applied Optics from Imperial College, London, and a Ph.D. from the University of Southampton in 1992. He joined Cranfield University as a research fellow in 1993, where he developed his interest in the development and application of optical instrumentation and sensors. As a Reader, he leads the optical fibre sensing activity in the Department of Engineering Photonics, with a focus on sensing chemical and physical parameters, and on deploying instrumentation in real world environments, ranging from foundation piles, railways, and aerospace structures to superconducting magnets.

Dr Frank Davis is currently a lecturer within the Centre for Biomedical Engineering at Cranfield University. He obtained a Ph.D. from Lancaster University in 1991 and worked at the Universities of Manchester and Sheffield, as well as a term in industry working for Gillette UKRDL. In 2002 he took up a post at Cranfield. His research interests include macrocyclic chemistry, ultrathin films, conductive polymers and the use of ultrasonic ablation to construct microelectrode arrays and biosensors. He is a co-author of *Macrocycles - Construction, Chemistry and Nanotechnology Applications*.

Professor Higson is Professor of Bio and Electroanalysis at Cranfield University and is Head of The Cranfield Biotechnology Centre. Professor Higson's career to date has spanned academic departments of Chemistry, Medicine, Biotechnology, Materials Science and Health - and this is reflected in the research interests of his group. Professor Higson's current research interests are primarily focused towards practical implementation of electro-analytical science and analytical biochemistry for biomedical, environmental and industrial process control applications. Professor Higson is also author of a number of books including the flagship text, 'Analytical Chemistry', published by the Oxford University press: (ISBN: 0 19-8502893).

Professor Ralph P. Tatam B.Sc. (Hons), Ph.D., D.Sc., CSci, CEng, CPhys, FinstP, FSPiE, was appointed to a personal Chair in Engineering Photonics at Cranfield University in 1998. He graduated from Exeter University in 1981 (B.Sc. Physics and Chemistry) and was awarded a Ph.D. in Physics (1986) for work on dynamic Rayleigh-Brillouin laser light scattering studies of liquids. He worked as a Research Fellow and Lecturer in the Physics Laboratory at the University of Kent developing optical fiber sensors from 1985 to 1989. In 1989 he took up an appointment at Cranfield where he has established an internationally recognised research team investigating optical instrumentation and sensors applied to challenging measurement problems in engineering and more recently biomedical. In 2004 he was awarded a D.Sc. by Exeter University for his research contributions. He has over 400 publications in journals and conferences.